

# Notes

## New and Facile Synthesis of Symmetrical and Unsymmetrical Diaryl Chalcogenides Using Trivalent Organobismuth Derivatives

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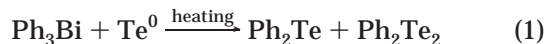
### Introduction

A number of syntheses of diaryl chalcogenide derivatives are already available,<sup>1</sup> but finding an easy and general method still remains a task of interest. Indeed, among the methods using lithium derivatives, Grignard reagents, and aryl diazonium and aryl iodonium salts, the early syntheses based on Ar<sub>2</sub>Hg as the aryl donor are still considered as the most general way to obtain symmetrical chalcogenides when reacting on elemental chalcogen<sup>2</sup> or unsymmetrical chalcogenides when reacting on a diaryl dichalcogenide.<sup>3</sup> However, these methods are often undesirable because of the high toxicity of the diarylmercury salts. Whereas numerous examples of bismuth–chalcogen interactions can be found either in mineralogy or in inorganic chemistry, very few accounts have been reported in the field of organic chemistry.<sup>4</sup> Wishing to examine the reactivity of triarylbismuthine derivatives toward chalcogens, we discovered two new reactions allowing the synthesis of symmetrical and unsymmetrical diaryl chalcogenides in good to high yield. Arylbismuthine derivatives are effective aryl donors on amines, enols, and phenols.<sup>5</sup> Now they prove to be efficient aryl donors in the preparation of diaryl chalcogenide derivatives.

### Results and Discussion

Heating triphenylbismuthine in the presence of tellurium metal (10 equiv) under reflux of benzene, toluene,

xylenes, mesitylene, or DMSO yields in a few hours a mixture of phenyl telluride and diphenyl telluride (eq 1).

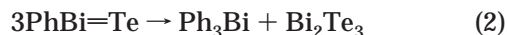


It was found that despite the long reaction time (24 h) in the case of benzene and toluene, some Ph<sub>3</sub>Bi still remained in solution. In xylenes and mesitylene, however, Ph<sub>3</sub>Bi was consumed in a reasonable amount of time (4.5 and 4 h, respectively) with a higher ratio in favor of diphenyl telluride (Ph<sub>2</sub>Te/Ph<sub>2</sub>Te<sub>2</sub> = 63/37) with the latter solvent. The ratio was somewhat reversed when heated under reflux of DMSO for 1 h (Ph<sub>2</sub>Te/Ph<sub>2</sub>Te<sub>2</sub> = 32/68).

Arylbismuthine derivatives are best known for the arylation of amines, enols, and phenols, which they are able to perform with high effectiveness.<sup>5</sup> However, only in a very few cases,<sup>6</sup> had it been possible to transfer to the functional organic group more than one aryl group out of the three that are available on the bismuth reagent. These arylation reactions could then be considered wasteful because two aryl groups were not utilized. However, in the present case, it must be noted that all the phenyl groups from the bismuth could be accounted for in the two products, Ph<sub>2</sub>Te<sub>2</sub> and Ph<sub>2</sub>Te.

Since phosphines react with elemental chalcogen in boiling toluene or at higher temperatures to yield stable species such as R<sub>3</sub>P=X,<sup>7</sup> it is reasonable to assume that the formation of the analogous bismuth(V) intermediate can constitute the first step in the reaction between Ph<sub>3</sub>Bi and Te<sup>0</sup> (Scheme 1). Ph<sub>3</sub>Bi=Te is likely to undergo rapid self-reduction to Ph<sub>2</sub>BiTePh. The oxidation/self-reduction process could repeat itself once or twice before a ligand coupling finally occurs, yielding the mono- or ditelluride derivative, respectively.

In both cases, PhBi=Te is generated, which could undergo a redistribution reaction (eq 2), common in bismuth chemistry.<sup>8,9</sup> Ph<sub>3</sub>Bi will therefore be regener-



ated, which accounts for the remarkable efficiency of the reaction. We suspect Bi<sub>2</sub>Te<sub>3</sub> to be the side product of the reaction. However, since the insoluble product that is formed during the reaction could not be separated from the excess of Te<sup>0</sup> used in the reaction, we have no evidence to support this hypothesis.

The procedure was successfully adapted to other triarylbismuthine derivatives (Table 1). The purification required no column chromatography. Instead, the products could be purified using a reduction–extraction

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<sup>‡</sup> In memory of Derek H. R. Barton (deceased March 16, 1998).

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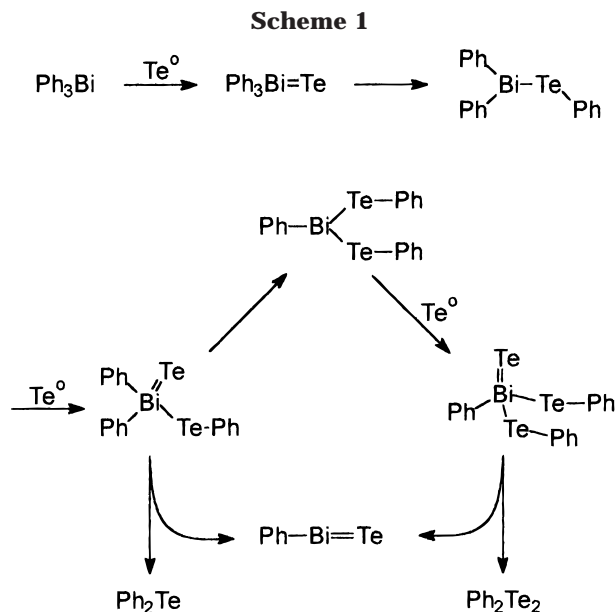
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**Table 1.** Ar<sub>3</sub>Bi + Te<sup>0</sup> (10 Equiv)<sup>a</sup>

aryl group	time (h)	Ar <sub>2</sub> Te <sub>2</sub> <sup>b</sup> (%)	Ar <sub>2</sub> Te <sup>b</sup> (%)
phenyl	12	34	66
mesityl <sup>c</sup>	24	<5	95
anisyl	3	<5	89
1-naphthyl	5	<5	93
TFMP <sup>d</sup>	30	61	39

<sup>a</sup> Reactions performed in mesitylene under reflux unless otherwise stated. <sup>b</sup> Isolated yields (given in phenyl groups transferred). <sup>c</sup> 5 equiv of Te<sup>0</sup> and reflux in toluene were sufficient in this reaction. <sup>d</sup> TFMP, *p*-(trifluoromethyl)phenyl. <sup>1</sup>H NMR yield.

**Table 2.** Ar<sub>3</sub>Bi + Se<sup>0</sup> (5 Equiv)<sup>a</sup>

aryl group	time (h)	Ar <sub>2</sub> Se <sub>2</sub> <sup>b</sup> (%)	Ar <sub>2</sub> Se <sup>b</sup> (%)
phenyl <sup>c</sup>	3	75	25
mesityl <sup>d</sup>	24		77
anisyl <sup>c</sup>	4	26	73
1-naphthyl	5	<5	94
TFMP	24	78	<5

<sup>a</sup> Reactions performed in mesitylene under reflux unless otherwise stated. <sup>b</sup> Isolated yields (given in aryl groups transferred). <sup>c</sup> Performed in xylenes under reflux. <sup>d</sup> Performed in toluene under reflux.

procedure.<sup>10</sup> However, in the case of TFMP the products could not be separated, and the ratio was determined by <sup>1</sup>H NMR, based upon the assignment of the spectral data reported in the literature.<sup>11,12</sup> In all cases but the latter, the monotelluride derivative was the major product.

Similar results were obtained with selenium (Table 2). Fewer equivalents of Se<sup>0</sup> were needed to bring the reaction to completion. Still, good yields of the monoselenide were obtained, albeit that the selectivity was generally lower than in the corresponding monotelluride.

Once again, a strong electron-withdrawing ligand had a dramatic effect on the product ratio. Indeed, only traces of the monoselenide were detected when the reaction was

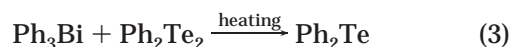
run with tris[*p*-(trifluoromethyl)phenyl]bismuthine. In most cases, the products were isolated by using only the reduction–extraction procedure described for the tellurides. But sometimes, column chromatography was necessary to purify the products further.

Comparing the tellurium and the selenium series, we observed that the ratio of Ar<sub>2</sub>Te/Ar<sub>2</sub>Te<sub>2</sub> is constantly higher or equal to the ratio of Ar<sub>2</sub>Se/Ar<sub>2</sub>Se<sub>2</sub> for one given ligand. This could be related to the different dissociation energies of the Bi–Te (230.8 ± 11.3 kJ·mol<sup>-1</sup>) and Bi–Se (279.0 ± 5.9 kJ·mol<sup>-1</sup>) bonds.<sup>13</sup> Also, in the tellurium series as well as in the selenium series the ratio of mono/dichalcogenide is obviously dependent on the electron-withdrawing force of the ligand transferred. Indeed, TFMP gives in both cases significantly less of the monochalcogenide than mesityl or anisyl ligands.

As discussed previously, the ratio of Ph<sub>2</sub>Te<sub>2</sub>/Ph<sub>2</sub>Te was reversed by changing the solvent to refluxing DMSO. Consequently, we were interested in exploring the possibility of controlling the ratio of mono- and dichalcogenide by modifying the reaction conditions. To see if Ph<sub>2</sub>Te<sub>2</sub> was produced from Ph<sub>2</sub>Te or vice versa, a simple kinetic experiment was carried out. The results show that Ph<sub>2</sub>Te<sub>2</sub> and Ph<sub>2</sub>Te appear to be formed simultaneously and that the ratio of these two products remains constant after Ph<sub>3</sub>Bi was consumed.

Nevertheless, it is known that Ph<sub>2</sub>Te<sub>2</sub> can lose a tellurium atom and yield Ph<sub>2</sub>Te when heated under high temperature.<sup>14</sup> However, a blank experiment showed that, under the reaction conditions, only 8% of Ph<sub>2</sub>Te could be isolated after 24 h, and 92% of the starting Ph<sub>2</sub>Te<sub>2</sub> was recovered. Clearly, the telluride does not arise from the thermolysis of the ditelluride.

Another pathway could explain the formation of Ph<sub>2</sub>Te as a secondary product from Ph<sub>2</sub>Te<sub>2</sub>. We studied the possibility of the reaction between Ph<sub>2</sub>Te<sub>2</sub> and Ph<sub>3</sub>Bi (eq 3). We found that the reaction occurs slowly (48 h in mesitylene under reflux for completion) and, therefore, can be disregarded as the main pathway for producing Ph<sub>2</sub>Te.



However, it was interesting to note that more than 1 equiv of Ph<sub>2</sub>Te, compared to Ph<sub>2</sub>Te<sub>2</sub>, was obtained in this reaction. Thus, Ph<sub>2</sub>Te is not produced by simple detellurization of Ph<sub>2</sub>Te<sub>2</sub> as is the case with copper,<sup>15</sup> nickel,<sup>16</sup> palladium,<sup>17</sup> and even cobalt.<sup>18</sup> It is more likely that the two moieties of the ditelluride are used in this reaction. Therefore, from one diphenyl ditelluride, two arylphenyl telluride derivatives are expected (eq 4).

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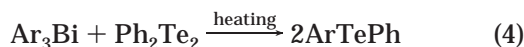
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(10) The separation of Ph<sub>2</sub>Te<sub>2</sub> and Ph<sub>2</sub>Te could not be performed by column chromatography due to their similar R<sub>f</sub> values. However, Ph<sub>2</sub>Te<sub>2</sub> can be extracted under its reduced form as phenyl telluride anion, allowing a quantitative separation and recovery of the two derivatives Ph<sub>2</sub>Te<sub>2</sub> and Ph<sub>2</sub>Te.



This would provide a new route for the synthesis of unsymmetrical tellurides and selenides simply if the aryl groups borne by the chalcogen and bismuth are different (Table 3).

As predicted, the reaction between diphenyl ditelluride or diphenyl diselenide with different triarylbismuthine species gave the unsymmetrical chalcogenides in good to high yield. No real difference in the yields can be observed when the reaction was performed either on the ditelluride or on the diselenide. Furthermore, as mentioned previously, from one  $\text{Ph}_2\text{X}_2$ , two  $\text{ArXPh}$  are produced, making this a particularly efficient reaction. As was mostly the case in Tables 1 and 2, mesitylene was again the preferred solvent. The choice of a lower boiling solvent like toluene when trimesitylbismuthine is used was driven by the fact that a complicated mixture of products was obtained when the reaction was performed at a higher temperature.

The reactions carried out in mesitylene had to be performed under argon atmosphere as we noticed that, otherwise, a significant amount of the solvent was air-oxidized to yield 3,5-dimethylbenzaldehyde.

### Conclusion

Two new and original reactions for the synthesis of symmetrical and unsymmetrical chalcogenides have been developed. Once again, triarylbismuthine reagents prove to be very efficient aryl donors. Their stability to moisture and air, as well as their low toxicity, give them a real advantage over most other aryl donors. Even though the results were not satisfactory in the particular case of the symmetrical derivatives bearing a strong electron-withdrawing ligand, this new procedure is easy and general enough to be compared to the well-known mercury salt method.

### Experimental Section

Melting points are uncorrected.  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{19}\text{F}$  NMR were recorded (300, 75, and 282 MHz, respectively) with  $\text{Me}_4\text{Si}$ , residual solvent peak, and  $\text{CFCl}_3$  as respective internal references. Elemental analyses were performed by Atlantic Microlab, Inc., Norcross, GA.  $\text{Ph}_3\text{Bi}$ ,  $\text{Ph}_2\text{Te}_2$ ,  $\text{Ph}_2\text{Se}_2$ , tellurium powder (200 mesh), and selenium powder (100 mesh) were purchased from Aldrich Chemical Co. Trimesitylbismuthine,<sup>9</sup> tris[*p*-anisyl]bismuthine<sup>19</sup> and tris[1-naphthyl]bismuthine<sup>20</sup> were synthesized following the literature procedures. The typical procedure is given for tris[*p*-(trifluoromethyl)phenyl]bismuthine<sup>21</sup> mentioned twice in the literature but for which no physical data have been given.

**Tris[*p*-(trifluoromethyl)phenyl]bismuthine.** A solution of 1-bromo-4-(trifluoromethyl)benzene (7.30 mL, 52 mMol) in dry THF (100 mL) was added to magnesium turnings (2.0 g, 83 mMol) under argon. The solution was transferred via cannula to a solution of  $\text{BiCl}_3$  (5 g, 15.86 mMol) in dry THF (50 mL). After the addition, the solution was quenched with methanol, and the volatiles were evaporated. The residue was taken in hexanes, and the solution was filtered and concentrated to yield the crude product. Crystallization from hexanes afforded the tris[*p*-(trifluoromethyl)phenyl]bismuthine as colorless plates (7.71 g, 75% yield): mp 145–146 °C;  $^1\text{H}$  NMR  $\delta$  7.65 (6H, m), 7.85

**Table 3.  $\text{Ar}_3\text{Bi} + \text{Ph}_2\text{X}_2$  (1 Equiv)<sup>a</sup>**

aryl group	X	time (h)	Ar-X-Ph <sup>b</sup> (%)
phenyl	Te	48	88
mesityl <sup>c</sup>	Te	48	95
anisyl	Te	48	74
1-naphthyl	Te	12	71
TFMP	Te	12	62
phenyl	Se	24	76
mesityl <sup>c</sup>	Se	24	62
anisyl	Se	48	62
1-naphthyl	Se	24	89
TFMP	Se	48	65

<sup>a</sup> Reactions performed in mesitylene under reflux unless otherwise stated. <sup>b</sup> Isolated yields (calculated with respect to the PhX moiety). <sup>c</sup> Performed in xylenes under reflux.

(6H, m);  $^{13}\text{C}$  NMR  $\delta$  118.71, 122.32, 125.93, 127.26, 127.31, 127.36, 127.41, 129.54, 129.90, 130.33, 130.77, 131.20, 137.76;  $^{19}\text{F}$  NMR  $\delta$  -63.38. Anal. Calcd for  $\text{C}_{21}\text{H}_{12}\text{BiF}_9$ : C, 39.15; H, 1.88. Found: C, 39.06; H, 1.87.

**Reaction between  $\text{Ar}_3\text{Bi}$  and  $\text{Te}^0$  or  $\text{Se}^0$ .** A typical procedure is given for  $\text{Ph}_3\text{Bi}$  and  $\text{Te}^0$ .  $\text{Ph}_3\text{Bi}$  (220 mg, 0.5 mMol) and  $\text{Te}^0$  (638 mg, 5 mMol) were stirred together in mesitylene (5 mL) under reflux for 12 h. After cooling, filtration, and evaporation of the solvent under oil pump vacuum, the residue was taken in THF (15 mL) and water (50 mL). A large excess of  $\text{NaBH}_4$  was added until the red color was completely discharged. Rapid extraction with ether (50 mL) afforded after drying ( $\text{MgSO}_4$ ) the pure diphenyl telluride (139 mg, 66%) as a colorless oil:<sup>22</sup>  $^1\text{H}$  NMR  $\delta$  7.20 ppm (3H, m), 7.70 ppm (2H, dd,  $J_1 = 8.1$  Hz,  $J_2 = 1.5$  Hz);  $^{13}\text{C}$  NMR  $\delta$  127.81, 129.48, 137.96; MS *m/e* 282 ( $\text{M}^+$ ).  $\text{HCl}$  (3 N) was added to the aqueous layer until no more  $\text{H}_2$  was released. Extraction with ether (50 mL), drying ( $\text{MgSO}_4$ ), and evaporation of the solvent afforded pure diphenyl ditelluride (104 mg, 34%) as a red solid:<sup>23</sup> mp 62–65 °C;  $^1\text{H}$  NMR  $\delta$  7.20 (3H, m), 7.80 (2H, dd,  $J_1 = 8.1$  Hz,  $J_2 = 1.5$  Hz);  $^{13}\text{C}$  NMR  $\delta$  128.06, 129.27, 137.60; MS *m/e* 409 ( $\text{M}^+$ ).

**Bis[1-naphthyl] telluride:** mp 108–109 °C (lit.<sup>24</sup> mp 126.5 °C, lit.<sup>25</sup> 123–126 °C);  $^1\text{H}$  NMR  $\delta$  7.22 (1H, m), 7.50 (2H, m), 7.80 (3H, m), 8.15 (1H, m);  $^{13}\text{C}$  NMR  $\delta$  126.30, 126.60, 126.97, 128.82, 129.22, 131.27, 133.78, 138.01; MS *m/e* 384 ( $\text{M}^+$ ). Anal. Calcd for  $\text{C}_{20}\text{H}_{14}\text{Te}$ : C, 62.90; H, 3.69. Found: C, 62.75; H, 3.64.

**Bis[1-naphthyl] selenide:** mp 106–107 °C (lit.<sup>24</sup> mp 114 °C);  $^1\text{H}$  NMR  $\delta$  7.28 (1H, t,  $J = 7.7$  Hz), 7.52 (3H, m), 7.79 (1H, d,  $J = 8.1$  Hz), 7.86 (1H, m), 8.34 (1H, m);  $^{13}\text{C}$  NMR  $\delta$  126.09, 126.33, 126.81, 127.09, 128.40, 128.61, 132.17, 134.11; MS *m/e* 334 ( $\text{M}^+$ ). Anal. Calcd for  $\text{C}_{20}\text{H}_{14}\text{Se}$ : C, 72.08; H, 4.23. Found: C, 72.05; H, 4.27.

**Reaction between  $\text{Ar}_3\text{Bi}$  and  $\text{Ph}_2\text{X}_2$ .** A typical procedure is described for  $\text{Mes}_3\text{Bi}$  and  $\text{Ph}_2\text{Te}_2$ .  $\text{Mes}_3\text{Bi}$  (283 mg, 0.5 mMol) and  $\text{Ph}_2\text{Te}_2$  (205 mg, 0.5 mMol) were stirred in xylenes (5 mL) under reflux until the red color was discharged (48 h). After filtration, evaporation of the volatiles, and column chromatography on silica gel (eluent: hexanes), mesitylphenyl telluride was isolated as a solid (307 mg, 95% yield): mp 27–29 °C;  $^1\text{H}$  NMR  $\delta$  2.31 (3H, s), 2.54 (6H, s), 7.01 (2H, s), 7.12 (3H, m), 7.32 (2H, m);  $^{13}\text{C}$  NMR  $\delta$  21.00, 29.50, 116.27, 118.28, 126.53, 127.57, 129.34, 134.59, 139.41, 145.45; HRMS calcd for  $\text{C}_{15}\text{H}_{16}\text{Te}$  326.0314, found: 326.0324. Anal. Calcd for  $\text{C}_{15}\text{H}_{16}\text{Te}$ : C, 55.63; H, 4.98. Found: C, 55.85; H, 5.00.

***p*-(Trifluoromethyl)phenyl phenyl telluride:** oil;  $^1\text{H}$  NMR  $\delta$  7.28 (2H, m), 7.40 (3H, m), 7.66 (2H, m), 7.80 (2H, m);  $^{13}\text{C}$  NMR  $\delta$  125.84, 125.89, 128.70, 129.84, 136.42, 138.02, 139.41;  $^{19}\text{F}$  NMR  $\delta$  -63.13; HRMS calcd for  $\text{C}_{13}\text{H}_9\text{F}_3\text{Te}$ : 351.9719, found: 351.9746. Anal. Calcd for  $\text{C}_{13}\text{H}_9\text{F}_3\text{Te}$ : C, 44.64; H, 2.59. Found: C, 44.84; H, 2.83.

***p*-(Trifluoromethyl)phenyl phenyl selenide:** oil;  $^1\text{H}$  NMR  $\delta$  7.35 (3H, m), 7.44 (4H, m), 7.57 (2H, m);  $^{13}\text{C}$  NMR  $\delta$  122.30,

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125.35, 125.40, 125.82, 125.87, 125.91, 125.97, 128.24, 128.52, 128.75, 129.71, 129.78, 130.95, 131.03, 134.83, 135.59, 137.21, 137.79;  $^{19}\text{F}$  NMR  $\delta$  -63.13; HRMS calcd for  $\text{C}_{13}\text{H}_9\text{F}_3\text{Se}$  301.9822, found: 301.9831.

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**Supporting Information Available:** Melting point and  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{19}\text{F}$  (when applicable) NMR data for all compounds except bis[*p*-(trifluoromethyl)phenyl] telluride and bis[*p*-(trifluoromethyl)phenyl] ditelluride, for which only  $^1\text{H}$  NMR data are available. Tables containing the optimization of the reaction conditions and the kinetic experiment. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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